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AN IMPROVED TECHNIQUE FOR FREEZE SAMPLING STREAMBED SEDIMENTS

by

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ABSTRACT

Equipment and operational improvements were made in the technique of freeze sampling stream sediments. The sampling procedure is routine, reproducible, and provides high quality samples. The equipment is now lighter, more reliable, and safer to use. The sediment sample obtained represents an undisturbed uniform vertical profile containing the bed material and water. Samples can be taken in deep water, through ice, and at other difficult locations. The equipment costs about $250.

KEYWORDS: Stream sampling, sedimentation.

INTRODUCTION

Freeze sampling (Walkotten 1973) provides the ability to sample in rocky streams, deep water, and arctic winter conditions (Lotspeich and Schallock 1974). The need to determine subtle environmental effects of sediment addition to salmon producing streams led to development and improvement of the freeze-sampling technique.

The technique provides an undisturbed sample with minimum disturbance of the sample area. The sample contains organic and inorganic material and intragravel water and retains sediment stratification.

Laboratory evaluation of this improved method compared with two other sampling methods[1] (McNeil and

[1]*Effects of land use in salmon production. Completion report, Commercial Fisheries and Development Act. Project No. 5-8-R and 5-19-R. Alaska Department of Fish and Game, Juneau. 1970.*

Ahnell 1960) in gravel of known composition, showed the freeze method to be the most accurate.[2]

Needed improvements in the original freeze-sampling technique became apparent as experience was gained with this method. It was difficult to control the high pressure liquid CO_2 accurately with conventional valves. This resulted in variable CO_2 use and plugging. Single point release of CO_2 also resulted in a "drop" shaped sample introducing possible bias. Modifications in the method were primarily concerned with improving sample quality. The use of an aluminum fire extinguisher for a supply tank reduces weight of the system. These changes provide the ability to closely meter the CO_2 at a known rate over time. The result is a reproducible testing procedure.

The sample probes are made from standard 3/4-inch harddrawn, type "M" copper pipe, a 3/4-inch brass or copper four-way cross fitting, and a tip machined from tool steel. The parts are soldered or brazed together.

The manifold parts are now commercially available (appendix B) and constructed to our specifications. Additionally, 4-foot-long manifold extension tubes are available if required. The filters are also available from commercial sources. Upon assembly, all nozzles and fittings on the manifold must be sealed with a thread sealer. This causes some difficulty when disassembling for repair or maintenance, but sealing is necessary to eliminate leaks at operating pressures.

[2]*Koski, K. Victor, and William J. Walkotten. Unpublished data on file at the Forestry Sciences Laboratory, Juneau.*

DESCRIPTION

Sampling is accomplished by freezing the substrate to a copper pipe probe inserted into the streambed (fig. 1). The rigid, thinwall copper pipe is mechanically strong and thermally conductive. Liquid CO_2 is released to atmospheric pressure from the manifold assembly placed inside the probe. The manifold distributes the CO_2 through metering nozzles over an area inside the probe that rapidly removes heat from the surrounding area in an even pattern. The manifold nozzles meter liquid CO_2 through #97 drill size (0.0057-inch) holes at a rate of about 0.2 lb/min per nozzle. The pressure drop and vaporization of CO_2 at the nozzle orifice causes rapid heat removal from the surrounding water and sediments, quickly freezing a core of sediment.

Operationally, the system is simpler and safer because a valve and some fittings are eliminated; and full pressure operation lets the entire system except the copper probe operate at ambient temperature. This greatly reduces the chance of component damage due to extreme thermal shock, keeps hoses flexible, reduces handling problems, and allows smaller size hose and fittings to be used. The addition of a 10-micron inline filter reduces the chance of plugging while sampling. This results in a smoother more reliable procedure with less maintenance. The elimination of a single point outlet for high pressure CO_2 lessens the chance of forcing the delivery manifold out of the probe.

The lower thermal conductivity of steel used in the probe tip causes a sharp cutoff of freezing at the bottom of the sample. This gives a more uniform shaped sample that is less biased by differences in size over depth.

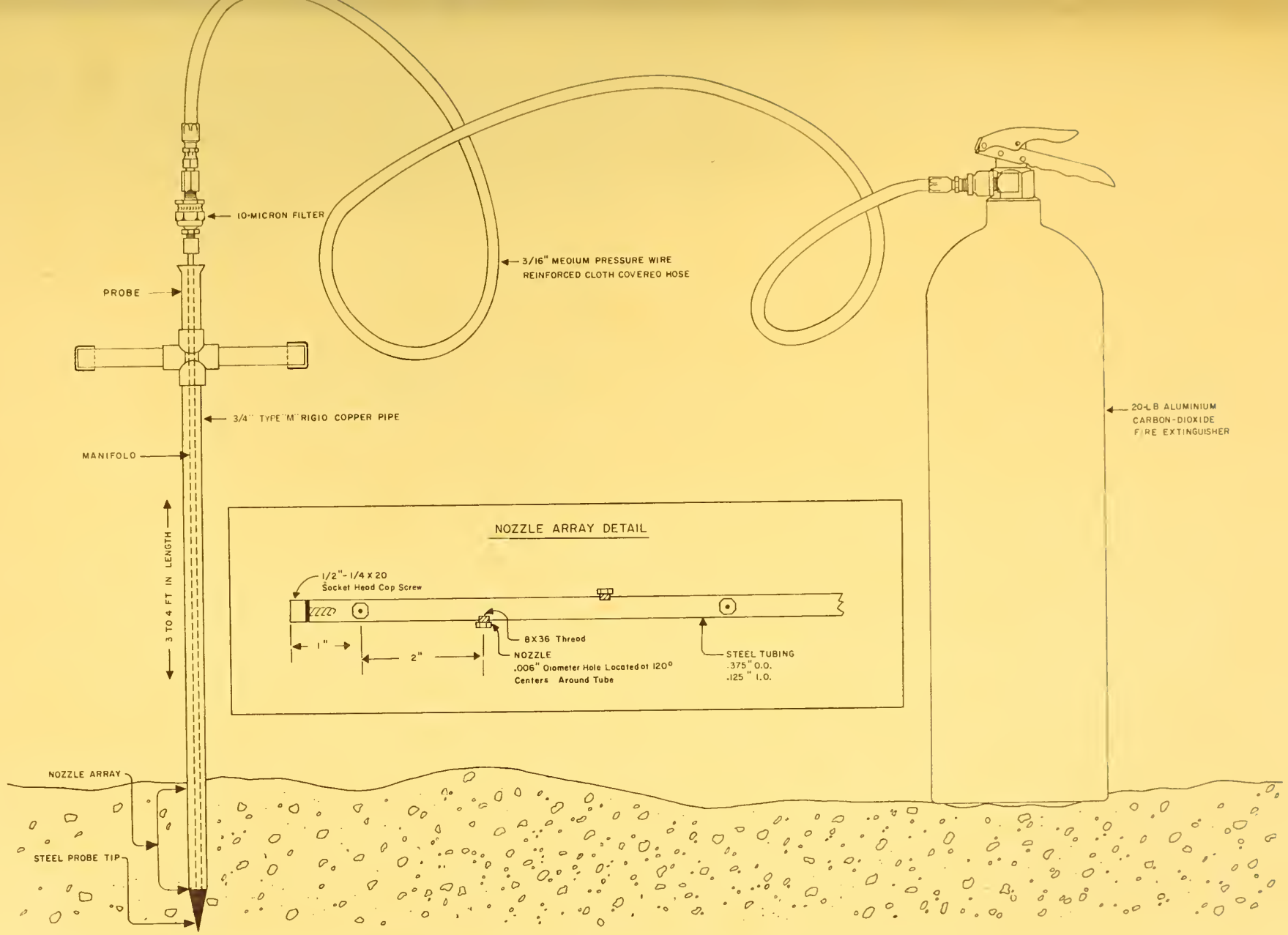

Figure 1.--Freeze sampling equipment showing probe, manifold, nozzle array detail, and CO_2 delivery system. The nozzles are located to release CO_2 below the gravel surface inside the copper probe.

PROBE AND MANIFOLD ASSEMBLY

The copper pipe probe (fig. 2) is made from standard 3/4-inch type "M" rigid copper pipe and fittings soldered together. A flared top is recommended to reduce the chance of shearing nozzles from the manifold when inserting it into the probe. The steel tip is machined for a tight fit inside the pipe and soldered in place.

The assembly of the manifold parts is important. The number of nozzles is determined by the chosen depth of the sample desired. Nozzle spacing on the manifold tube is determined by the area that a single nozzle can effectively freeze without noticeable change in the shape of the sediment sample. Testing showed this spacing to be about 2 inches. Nozzles are placed in the manifold from the bottom up to a point 2 to 3 inches below the streambed gravel surface. For example, four nozzles would be placed in the manifold for a 10-inch-deep sample,

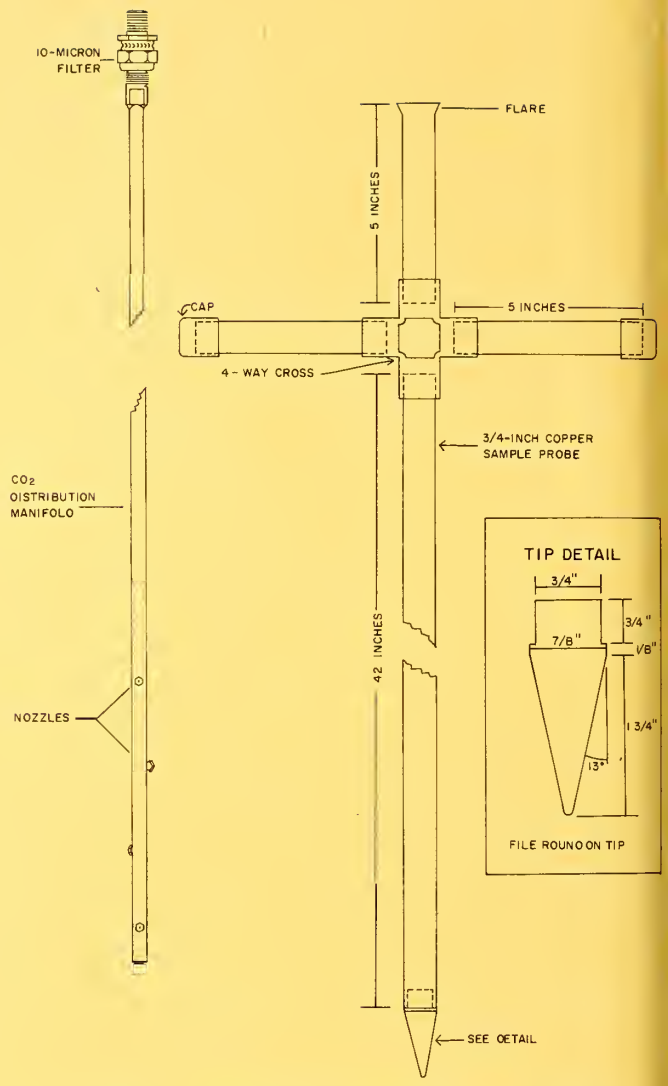

Figure 2.--Sampling probe and manifold assembly.

and the rest of the holes would be filled with nozzle blanks. CO_2 use is about 0.2 lb/nozzle per minute so the above manifold assembly would release about 0.8 lb of CO_2 per minute of sampling.

After the sample site is selected, the copper probe is pushed into the streambed, the manifold assembly is inserted into the probe, and the CO_2 is turned on. Timing is all that is necessary to freeze the desired sample. The CO_2 is stopped, the manifold removed from the probe, and the probe and sample are pulled from the streambed. A good quality sample can be obtained in 4 minutes (table 1).

FIELD APPLICATION

This sampling method allows great flexibility in selecting sample locations and allows samples to be taken over a variety of bottom types, current velocities, and depths. This system allows the operator to either select random sites or utilize a carefully designed method of sampling.

If several copper probes are placed in the streambed prior to sampling, the manifold can be moved from one to another without turning the CO_2 off. A frozen sample can be extracted after the manifold is moved to the next probe and timing begins. Some preplanning is necessary to prevent a mixup of samples but total time spent sampling is reduced by this routine.

Table 1--*Timed CO_2 sampling tests using four nozzles at a 10-inch sample depth in laboratory blended gravel*

N = 3

Freeze time (minutes)	Mean diameter of freeze sample	Mean sample weight	Mean CO_2 used per sample	CO_2 used per kg sample	Gravel percentage passing through a 3.36 mm-sieve	
	cm	kg	- - - - [1/] lb - - - - -		Mean	Sample standard error
2	5.84	1.095	2.0	1.84	18.5	6.1
4	7.62	1.586	3.3	2.16	21.7	2.0
6	7.87	2.052	4.9	2.38	23.2	2.5
8	8.89	2.534	6.1	2.42	22.4	2.7
10	9.14	2.809	7.6	2.74	21.0	2.4
15	10.92	2.688	11.6	3.15	24.7	2.7

[1/] Carbon dioxide is bottled and sold on the commercial market by weight in pounds and is reported here in pounds used per sample for cost comparison purposes with local prices.

A hoisting or leverage device may be necessary to loosen the frozen core from the streambed if large samples are needed or if the depth of sediments is more than about 12 inches. A satisfactory lightweight tripod hoist (Gary 1976) can be fabricated using aluminum pipe and a small hand winch. It is necessary to weld a 4-inch-square plate on the bottom of the legs to prevent them from sinking into the gravel. Once the sample is broken loose from the streambed, it can usually be removed by lifting.

It is essential to supply the nozzles with liquid CO_2. A fire extinguisher bottle and some other storage cylinders have an internal tube extending to the bottom of the container so the discharge is liquid under pressure in the upright position. If cylinders with the internal tube cannot be obtained, the liquid CO_2 can be removed by inverting the tank.

ASSEMBLY AND USE

Assembly and Operation: If possible, set the CO_2 cylinder on level ground. Remove the safety pin; and while holding the cylinder firmly with the outlet pointed in a safe direction, tap the discharge lever to release a small amount of CO_2. This clears the valve of accumulated dirt and condensation. Apply thread lubricant on the hose fittings and attach hose and manifold assembly to the cylinder. Carefully tighten all fittings with a wrench. Holding the manifold away from the operator, turn on the CO_2 and check for leakage and plugged nozzles. If everything checks, carefully insert the manifold into the previously located copper probe, tie down with a nylon cord, and start timing.

The copper sample probe is placed in a selected location by pushing it to the desired depth by hand or by driving it down with a steel rod placed inside. The rod is driven with a hammer against the probe tip. The driving method of probe placement works unless the probe strikes a large rock, and then forcing the probe will only damage it. For sampling, attach about 10 inches of filament tape to the top of the discharge lever on the CO_2 cylinder, open the discharge valve, and tape it down. Sample timing should start when "snow" appears above the top of the probe which indicate that the liquid CO_2 is flowing through the nozzles and the freezing process has begun. After 4 minutes, release the tape holding the discharge lever down or switch the manifold to another sampling probe. Since the CO_2 discharge is through metering nozzles, the manifold can be removed while still operating and placed in another probe. This should be done carefully, and the operator must wear eye protection.

Remove the frozen sample probe from the streambed, using a lifting device if necessary, and place it in a plastic bag for thawing. Do not use a high temperature heat source such as a propane torch to heat the probe in an attempt to accelerate the thawing process. This anneals the rigid copper pipe so it will bend in use. Hot water poured into the probe works best if thawing must be hurried. Be sure to take notes and label samples at this time to prevent later mixups. Generally, four samples can be taken with a 20-lb cylinder, but is must be remembered that successful freezing will take place only when liquid CO_2 is delivered to the probe. When in doubt about the supply, the cylinder can be weighed in the field with a spring scale. A full 20-pound aluminum extinguisher bottle weighs 46 to 47 pounds so estimates on available supply can be made. Each nozzle releases CO_2 at a rate of about 0.2 lb per minute, and even a small leak in the system releases a large quantity of CO_2 compared to the nozzles. It is advisable to check for leaks and plugged nozzles whenever the manifold is placed in or removed from the probe.

Field sampling requires some accessory equipment along with the basic sampling gear. Experience has shown that a basic set of tools, parts and accessories assembled into a kit is necessary for field repairs and maintenance. Appendix A shows the recommended contents of such a kit.

MAINTENANCE AND SAFETY PRECAUTIONS

Use, transport, assembly, and operation of the equipment causes some damage and a certain amount of maintenance is always necessary to prevent loss of time. Since the CO_2 is in cylinders under pressure (830 PSIG at 70 °F), a potential hazard should be recognized; and although the cylinders can withstand normal handling, any chance of abuse (dropping, stripping threads, no safety caps on outlets, etc.) must be eliminated and questionable equipment should not be used.

Each CO_2 cylinder should be plumbed with steel fittings so that the hose can be changed without removing the pipe fittings from the aluminum valve housing. This prevents thread damage to the valve and is faster than removing the fittings. A pipe plug should be attached to each cylinder for plugging the fitting when the hose is removed.

It is only necessary to remove the hose from the CO_2 cylinder and manifold to prepare the sampler for shipment. It must be remembered that the CO_2 cylinders are still under pressure, and the outlet port needs to be plugged and the valve safety pin needs to be taped in place to prevent accidental discharge. Never bleed all the pressure from the cylinder as this may loosen the valve seals and cause leakage upon refill.

Some routine maintenance is necessary after about 50 samples. Remove the inline filter from the hose and manifold if plugging becomes a problem. Install the filter on a CO_2 cylinder using a 1/4-inch short pipe nipple with the flow arrow pointing toward the CO_2 cylinder to reverse normal direction of flow. Clean the filter by grasping the cylinder firmly and expelling a small amount of CO_2 through the filter. The filter can then be reinstalled on the manifold using a small amount of thread sealer on the fittings.

Periodically, and after any repair, remove the cap screw in the manifold and blow the hose and manifold clean. If nozzles plug and cannot be cleaned, remove them and check the screens. Plugged nozzles and screens must be replaced and the entire system blown clean. Experience has shown that badly contaminated CO_2 containing rust, dirt, water, and oil can occur. In that case, cylinder and valves must be cleaned. The supplier should be notified and should clean the cylinders. The recommended hose has removable fittings so repairs can be made in the field. If a leak develops, the fittings can be removed, the hose cut with a fine-tooth hacksaw, and the fittings replaced.

Nozzles can be broken off unless they are carefully removed with a well-fitting 7/32-inch socket wrench. The removable screen inside the nozzle can be replaced with a small rounded device such as a ballpoint pen tip. If a nozzle is broken off, it usually cannot be removed with an ezyout and must be drilled out with a #29 bit and the threads cleared with a #8x36 thread tap. This must be carefully done to prevent permanent damage to the manifold tube. A small amount of thread sealer is placed on all reassembled parts. The thread sealer will prevent leakage after it sets. Do not overtighten and strip the threads on the brass nozzles.

Repair or replace the copper probe if it becomes bent or leaks water. Normal use rarely causes damage to the probe, but forcing penetration with a driving rod can drive the point off the end of the tube, and cause the probe to

bend. If driving is used for a placement method, more damage should be expected and additional parts should be available.

In general, the sampling gear should be kept as clean and dry as possible, especially the internal parts of the CO_2 system. Care in handling equipment during shipment and sampling will reduce maintenance problems.

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APPENDIX A

RECOMMENDED FIELD SAMPLING AND MAINTENANCE EQUIPMENT LIST
FOR CO_2 SAMPLER

Sampler Equipment

3 ea. copper sample probes
1 ea. #M0297 CO_2 metering manifold assembly (CO_2 probe)
1 ea. hose assembly
2 ea. or more 20-lb. aluminum fire extinguisher bottles
1 pr. goggles or safety glasses
1 ea. probe driving rod, 5/8-inch diam. x 54 inches long
1 ea. driving hammer
1 ea. stopwatch
1 pr. gloves
1 roll filament tape
3- to 4-foot-length 1/8-inch nylon cord

General Tools and Equipment

1 ea. tool box
1 ea. 12-inch adjustable wrench
2 ea. 8- or 10-inch adjustable wrenches
1 ea. 4-inch adjustable wrench
1 ea. pocket knife
1 ea. 8-inch pipe wrench
1 ea. 50-lb spring scale
1 ea. pack board for transporting CO_2 cylinders
1 ea. pack for transporting sampling and maintenance gear
1 ea. tripod assembly (optional)

Maintenance Equipment

1 ea. #97 drill for cleaning nozzles
1 ea. #29 drill bit for drilling out broken nozzles
1 ea. 7/32-inch socket wrench
1 ea. 8-36 taper, plug & bottom thread tap set
1 ea. #1 screw extractor
1 ea. hacksaw with fine-tooth blade (32 per inch)
1 ea. large vice grips
1 ea. tube thread sealer
1 ea. tube thread lubricant
1 ea. small hand drill
2 ea. 1/4-inch short pipe nipples
1 ea. Gates 4C54RPM male hose fitting
1 ea. Gates 4C54RFSX fitting
1 ea. Gates 4F-4SMS adapter
12 ea. Modern MFG Co. M02981 modified nozzles
6 ea. Modern MFG Co. M02982 modified nozzle blanks
24 ea. Modern MFG Co. nozzle screens
1 ea. nozzle screen inserter (ballpoint pen)
1 ea. propane torch soldering kit
Spare solder, flux, & steel wool
1 ea. spare steel probe tip
1 ea. Linde Model SG - 6110 inline filter

APPENDIX B

FREEZE SAMPLER SUPPLY SOURCES[3] - 1975 PRICES

CO_2 manifold, nozzles, and accessories. Approximate cost: $90
Price schedule for MO297 CO_2 probe manifold:

NUMBER	PRICE EACH
25 & OVER	$65.00
20-24	70.00
15-19	75.00
10-14	80.00
1-9	85.00
MO298-1 Modified nozzles W/.0057 orifice	1.50
MO298-2 Modified nozzle blank	.75
4-foot extension manifold tube	32.25

The manufacturer lists the manifold as "MO297 CO_2 PROBE" for supply purposes. This does not include the copper sample probe.

Modern Manufacturing Inc.
815 Houser Way N.
Renton, WA 98055

(206) 228-4500

Inline Filter Model SG-6110 $18

Union Carbide Corporation
Lindie Division
One California St.
San Francisco, CA 94111

(415) 765-1322

Hose & fittings 4C5 Gates hose 15 to 20 feet @ $.97/ft
4C5-4RPM male fitting 1 each @ $.60 each
4C5-4RFSX fitting 1 each @ $.80
4F-4JMS adapter 1 each @ $.74

Nebar Supply Company Inc.
430 Minor Ave. N.
Seattle, WA 98109

(206) 622-6292

20-lb aluminum fire extinguisher, approximate cost: $50

United Fire Extinguisher Sales & Services
4611 N.E. Union Ave.
Portland, OR 97211

(503) 288-5716

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[3]*Trade names mentioned are for convenience of reader and do not imply endorsement by the U.S. Department of Agriculture.*

4-way brass or copper cross, 3/4-inch $5.50

Alaskan Copper & Brass Company
3223 Sixth Ave. S.
Seattle, WA 98108

(206) 623-5800

8x36 thread taps	Approximate cost each:	$1.00
8x36 thread die	Approximate cost each:	$1.25
#97 drill bit	Approximate cost each:	$1.50
#29 drill bit	Approximate cost each:	$1.00

Sterling Tool Company
4202 6th Avenue S.
Seattle, WA 98108

(206) 623-6891

Miscellaneous copper pipe, pipe fittings, tool steel, thread sealer, 7/32-inch socket wrench, solder, tools, etc. From local suppliers.

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